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## PATENT SPECIFICATION

NO DRAWINGS

927,492



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Date of Application and filing Complete Specification: June 30, 1960.

No. 22992/60.

Application made in United States of America (No. 605,192) on Aug. 3, 1959.

Complete Specification Published: May 29, 1963.

Index at acceptance:—Classes 39(4), O; 37, E; 38(2), H(1:2:3B:3E:4:5B:5C2:6A:6B:8A); 39(1), S(1:3), S4(B:C:D:N:R:S); and 40(3), A5B4.

International Classification:—G21. (C09k. G01r. G08c. H01d).

### COMPLETE SPECIFICATION

#### Improvements in or relating to Magnetic Test Particles

I. JOHN DAYTON STEPHENS, a citizen of the

#### ERRATUM.

#### SPECIFICATION NO. 927,492

Page 1 in the heading number of Application made in United States of America should read "A31,031."

THE PATENT OFFICE,  
25th October, 1963

D 30039/1 (3) / 200 10/63 PL

20 testing methods for locating flaws in parts and structures of ferromagnetic materials, hereinafter sometimes referred to as "test bodies." Examples of such flaws are voids and discontinuities with or without surface openings, such as cracks, blow holes, forging laps, laminations, deep scratches, as well as occlusions of solid material which is non-magnetic or substantially less paramagnetic than the test body, such as slag stringers. In general such magnetic flux systems of non-destructive testing comprise the steps of creating a magnetic flux within, or passing a magnetic flux through, the test body. A flaw 30 in the test body will concentrate the magnetic flux in the area of the flaw and cause a powder of magnetic particles to adhere to the surface of the body at the point or line of the flaw; the particles, so adhered, thereby reveal the 35 existence and location of the flaw and will frequently give an indication of the size and nature of the flaw as well. These magnetic flux systems are used in the aircraft industry and other fields.

40 Extensive efforts have been made to improve the methods and testing materials as to sensitivity, effectiveness, ease, and efficiency of operation. The original magnetic flux systems employed magnetic particles that 45 were either dull, brownish red or black in

fluorescent and magnetic. The test body is then inspected in the substantial absence of visible light but under invisible fluorescogenous radiation, such as filtered ultraviolet light (black light). A small amount of visible violet light passing through the filter and reflecting from the test body gave the test body a dark blue or purple cast. The black light, however, caused the fluorescent magnetic particles adhering to the test body at the location of the flaws to emit visible light. The vastly increased contrast ratios between the fluorescent magnetic particles emitting visible light and the dark background of the test body greatly expedited the discovery of many flaw indications, especially in difficultly inspected locations such as the inside surface of coil springs or the interior of tubes that might otherwise pass without detection.

50 In actual practice both the nonfluorescent and the fluorescent systems of magnetic flux location require constant visual searching of the test bodies in order to locate the flaw indications. The necessity of inspecting visually every test body in a production batch consisting of numerous pieces has necessitated in many cases the use of a large number of operators. Furthermore, the monotonous and tedious searching of every piece for flaws can tire and render an operator inattentive, causing the operator to overlook critical flaw 55 60 65 70 75 80 85 90 95

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## COMPLETE SPECIFICATION

### Improvements in or relating to Magnetic Test Particles

I, JOHN DAYTON STEELE, a citizen of the United States of America, of 767, Union Commerce Building, Cleveland, Ohio, United States of America, do hereby declare the invention, for which I pray that a patent may be granted to me, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 The invention relates to radioactive magnetic inspection compositions and methods of making and utilizing such compositions.

15 Magnetic flux systems of nondestructive testing are, and have been for many years past, among the most widely and successfully used testing methods for locating flaws in parts and structures of ferromagnetic materials, hereinafter sometimes referred to as "test bodies." Examples of such flaws are voids and discontinuities with or without surface openings, such as cracks, blow holes, forging laps, laminations, deep scratches, as well as occlusions of solid material which is non-magnetic or substantially less paramagnetic

20 than the test body, such as slag stringers. In general such magnetic flux systems of non-destructive testing comprise the steps of creating a magnetic flux within, or passing a magnetic flux through, the test body. A flaw in the test body will concentrate the magnetic flux in the area of the flaw and cause a powder of magnetic particles to adhere to the surface of the body at the point or line of the flaw; the particles, so adhered, thereby reveal the

25 existence and location of the flaw and will frequently give an indication of the size and nature of the flaw as well. These magnetic flux systems are used in the aircraft industry and other fields.

30 Extensive efforts have been made to improve the methods and testing materials as to sensitivity, effectiveness, ease, and efficiency of operation. The original magnetic flux systems employed magnetic particles that

35 were either dull, brownish red or black in

colour and the particles attracted to the flaw were looked for visually under ordinary light.

The visibility of the flaw indications under these circumstances left much to be desired because of the frequently poor contrast between the particles and the metal background. Indeed, under visible light many indications were missed if the flaws were fine or minute and their indications correspondingly small.

A substantial improvement with respect to visibility was disclosed in U.S. Patent No. 2,267,999 and essentially comprised the use of a magnetic flux as described above, but with testing powders whose particles are both fluorescent and magnetic. The test body is then inspected in the substantial absence of visible light but under invisible fluorescogenous radiation, such as filtered ultraviolet light (black light). A small amount of visible violet light passing through the filter and reflecting from the test body gave the test body a dark blue or purple cast. The black light, however, caused the fluorescent magnetic particles adhering to the test body at the location of the flaws to emit visible light. The vastly increased contrast ratios between the fluorescent magnetic particles emitting visible light and the dark background of the test body greatly expedited the discovery of many flaw indications, especially in difficultly inspected locations such as the inside surface of coil springs or the interior of tubes that might otherwise pass without detection.

In actual practice both the nonfluorescent and the fluorescent systems of magnetic flux location require constant visual searching of the test bodies in order to locate the flaw indications. The necessity of inspecting visually every test body in a production batch consisting of numerous pieces has necessitated in many cases the use of a large number of operators. Furthermore, the monotonous and tedious searching of every piece for flaws can tire and render an operator inattentive, causing the operator to overlook critical flaw

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indications on the test bodies under the psychologically fatiguing condition of repetitively examining substantially identical parts or pieces.

5 To eliminate the chance for human error, attempts have been made to supplement or replace visual inspection of the test bodies with visible light responsive means, as with photoelectric cells which would respond to 10 the visible light emitted by the flaw indications in the fluorescent systems. To date, no such systems which would supplement or replace human visual inspection have proved sufficiently reliable. It is, therefore, an object 15 of this invention to provide a magnetic flux system of nondestructive testing which reduces the chance of human failure in the detection of flaws.

The present invention is based upon the 20 use (instead of, or conjointly with, the fluorescent indication secured by the fluorescent system of nondestructive testing) of a radioactive material, either to activate the fluorescent component of the fluorescent paramagnetic testing powders or to serve as the 25 indicating element of the powders. However, in any method of simply adding a radioactive material to the mixture of paramagnetic cores and adhesive materials to produce a fine 30 paramagnetic powder comprising a paramagnetic element, an indicating element, and an adhesive material joining the two, such a method necessitates working with radioactive materials which may be not only dangerous to 35 the workmen preparing the powders but may also dangerously contaminate the production machinery required in the production of the powders. Therefore, wholly apart from the danger to human life involved, the production 40 of radioactive paramagnetic testing powders has not been economically feasible because of the cost of the production equipment which would be then contaminated or endangered.

Basically the paramagnetic particles the 45 subject of this invention are made by adhering to paramagnetic cores a material that will readily absorb or react with a radioactive component, to bind the latter to the core, the adhesion taking place preferably 50 through the intermediate agency of an adhesive resin having the property of hardening in stages which allows the processing of the composite material into powders or slurries having the desired composition and 55 particle size characteristics.

According to the invention therefore we provide a paramagnetic radioactive powder comprising particles composed of one or more paramagnetic particles, a carrier material for 60 a radioactive material adhesively bonded to said paramagnetic particle(s) by means of an adhesive preferably by a synthetic resin, and a radioactive material bonded to an exposed surface of said carrier material by physical 65 or chemical bonds.

The word "paramagnetic" is used in this specification to describe substances having a magnetic permeability greater than 1, and is intended to include within its scope substances having a magnetic permeability many times greater than 1 which are commonly known as "ferromagnetic" substances. The paramagnetic particles of the powder according to the invention are preferably ferromagnetic.

The paramagnetic radioactive powder according to the present invention may also contain a visible light-emitting fluorescent material and/or colouring materials in addition to the carrier and radioactive materials. When a fluorescent material is present the radioactive material may be one which serves to activate the fluorescent material.

Among the paramagnetic particles that may be used are the magnetic iron oxides, both red and black, metallic iron prepared by the carbonyl process, aluminium, and any other material that is paramagnetic. (See Smithsonian Physical Tables," Fowle 1927, Seventh Edition). It must be borne in mind that the higher the magnetic susceptibility the better the material is as a magnetic core.

Adhesion of the carrier material (and in some cases the fluorescent material) to the magnetic core substance may be achieved by the lacquer method of the Switzer United States Patent No. 2,267,999 or by the method of British Patent No. 833,267 for "Magnetic Particles." The latter method employing epoxy type adhesive resins is the most satisfactory, although the use of other cross-linking or thermosetting resins such as phenol formaldehydes, urea formaldehydes, melamine formaldehydes, polyurethanes, and polyesters is quite feasible.

After the paramagnetic component, an adhesive or combining material, and a reactive or absorptive carrier material have been combined and reduced to the desired particle sizes (with or without fluorescent materials combined therewith), the particles are dispersed in a bath or medium in which radioactive materials are also dispersed. Said radioactive materials then become attached to the paramagnetic powder by adsorption or chemical reaction.

Thus, it will be noted that the phenomenon by which the radioactive materials become incorporated is essentially a surface treatment of the individual particles in the paramagnetic testing powders. Such incorporation will not contaminate the machinery for producing the paramagnetic powder nor is it necessary that the radioactive components be incorporated at the same place and time where and when the powders are formed. Instead, the paramagnetic powders may be produced and stored as a nonradioactive product and altered to their radioactive condition just before use. Further, because these powders are

essentially surface treated, after use both the paramagnetic radioactive powders themselves and the bodies tested therewith may be subjected to de-contaminating treatments, a step not heretofore feasible with previously proposed radioactive magnetic powders which were likely to have the radioactive component encased by an impervious resin resistant to a treatment which might otherwise attack and decontaminate the radioactive component. 70

The carrier material may, as indicated, be a purely absorptive or adsorptive material which mechanically carries the radioactive material. Such absorptive or adsorptive carrier material includes, for example, silica gel, diatomaceous earth, and activated clays, which may be reduced to pigmentary particle size and still retain their absorptive or adsorptive properties. Such products may be satisfactory, particularly when the radioactive material has a relatively short half-life and no problems are encountered in working with a solution or liquid dispersion of radioactive material of sufficient concentration to ensure that substantially all particles will exhibit adequate radioactivity after treatment and the half-life is sufficiently short to avoid serious disposal or decontamination problems, either of the spent paramagnetic radioactive 75

30 powders or in subsequent handling of test bodies to which the powder particles may cling or in which the powder particles may be entrapped after testing.

In general, however, we have found it preferable to incorporate the radioactive material in the paramagnetic testing powders by a chemical reaction between the radioactive material and the carrier material bonded to the paramagnetic cores. The chemical reaction involved may follow any one of the standard types of chemical reaction: simple addition ( $A + B = AB$ ), substitution ( $AC + B = AB + C$ ), and double decomposition and synthesis ( $AC + BD = AB + CD$ ). Of these 80

45 three types of reaction, the double decomposition type of reaction is usually more satisfactory by permitting the use of ion exchange or chelating materials. Such materials are characterized by an ability to extract exceedingly minute and dilute proportions of the radioactive material from liquid solutions or dispersions, in which the radioactive material may be carried prior to incorporation in the paramagnetic testing powders. Further, especially when using ion exchange materials, the reaction may often be reversed, aiding in decontaminating tested bodies or articles and extracting the radioactive component from the spent paramagnetic radioactive powders so 85

50 that the radioactive component is separated in a form permitting of convenient disposal.

Either during or after adhering the carrier material to the paramagnetic core particles, the massive aggregate produced is broken up 90

55 or ground into fine discrete particles of the average size desired. Grinding in water is satisfactory in most cases. The slurry that forms may be levigated or subjected to a settling process or a magnetic separation to remove any nonmagnetic material that breaks off during the grinding operation; for example, to remove a certain amount of brittle ion exchange material. Usually if the grinding is thorough, very little additional material will break off during subsequent use. Processing in water has an advantage that the ion-exchange resin or other adsorptive or chelating carrier material is properly wet through or swelled and is in a better condition to receive the radioactive substance. 95

60 Some of the paramagnetic powders containing ion-exchange resins may be treated chemically after the bonding and grinding steps are complete to convert them to the proper chemical form. Thus the polyacrylic acid type of ion-exchange resin is generally converted from its acid or carboxy form into a salt such as the sodium salt by treating the powders containing it with sodium hydroxide or some other alkali. This conversion is carried out either batchwise or in a column by contacting with aqueous alkali, then washing well with water.

The choice of ion-exchange resin or other absorptive or reactive carrier material depends in some cases upon the radioactive substance. With radioactive reagents of high specific activity an adsorptive or reactive carrier material of only relatively low capacity is required. In that case almost any carrier material will suffice that will not allow the radioactive material to be leached out when in contact with an aqueous or nonaqueous suspending medium. With radioactive reagents of low specific activity on the other hand, a carrier material of high capacity is required. For example, if the radioactive material is a cobalt chloride prepared by dissolving neutron-irradiated metallic cobalt in which only one cobalt atom in 100,000 is converted from  $Co^{59}$  to  $Co^{60}$ , an exchange capacity is required in the ion-exchange resin of at least 2 milliequivalents per gram of dry resin if only a single batchwise contact with the cobalt chloride solution is to be made. 100

65 Commercial ion-exchange resins, if they have been converted to the proper chemical forms, usually have ample ion-exchange capacity for the adsorption of practical amounts of radioactive ions. For example, the sulphonated cross-linked polystyrene type of cation exchange resin in the sodium form is suitable for most of the positive radioactive ions. This resin will efficiently take up most trivalent ions from dilute solution. Divalent ions and the larger monovalent ions are also fairly efficiently taken up in one batchwise contact followed by a water wash. Since the above resin is inexpensive and one of the most versatile of the cation-exchange resins, 105

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it is the preferred carrier material in many cases. The cross-linked acrylic acid type of resin, for example in its sodium form, is another cation-exchange resin of great value. It has the advantage of being sufficiently tough and flexible to show little breakdown during a long grinding operation on the bonded powder particles. Frequently no separation or settling step is required. Its affinity for cobalt ions, for example, is so strong that radioactive cobalt ions are taken up very efficiently in a single batchwise contact with a cobalt<sup>60</sup> chloride solution.

Many other ion-exchange resins are useful in particular instances. Thus caesium<sup>137</sup> ions are strongly taken up by sulphonated phenol-formaldehyde cation-exchange resins made by reacting formaldehyde with phenol *m*-sulphonic acid. Another cation-exchange resin of the formaldehyde type which is suitable for cobalt<sup>60</sup> is made by condensing formaldehyde with 5-resorcylic acid. Other cation-exchange resins which may be used as the carrier material include formaldehyde-phenol carboxylic acid resins. The radioactive positive metal ions used as the radioactive material with the cation-exchange resins preferably have an emission energy which is in excess of 50,000 electron volts.

Negative radioactive ions, such as sulphate ions derived from sulphur<sup>35</sup> are taken up from solution by anion exchange resins, such as certain quaternary ammonium derivatives of cross-linked polystyrene. The use of this type of resin and of the weakly basic ion-exchange resins extends the range of the radioactive paramagnetic powders to include many nonmetallic radionuclides and metallic radionuclides in their higher valence forms. Thus tantalum<sup>182</sup> may be retained by the basic resins as a tantalate ion, Ta<sub>2</sub>O<sub>6</sub> or Ta<sub>6</sub>O<sub>11</sub>. Examples of other radioactive materials providing negative ions are iodine<sup>131</sup> in the iodide form, sulphur<sup>35</sup> in the sulphide and sulphate forms, and phosphorus<sup>32</sup> in the phosphate form. Preferred radioactive negative ions are those having an emission energy in excess of 50,000 electron volts.

Ion-exchange resins for use as carrier materials in powders according to this invention may also have metal chelating properties. The polyacrylic acid type of resin already described probably owes much of its activity to the attraction of each polyvalent metal ion to separate but adjacent carboxyl groups to form nonplanar metal-containing rings. A deliberate insertion of metal-chelating groups is also possible. For example, cross-linked polystyrene resins, such as the copolymer of styrene and divinyl benzene may be given metal-chelating properties by substituting di-(carboxymethyl)-aminomethyl groups onto the benzene rings, for example, by reacting first with monochloromethyl ether then with a salt of imino diacetic acid. The tridentate chelating groups so formed confer the property of sequestering metal ions.

Ion exchange substances other than ion exchange resins can be used as the adsorbing carrier material. Natural zeolites, synthetic zeolites and certain clays have ion exchange characteristics and can be used with at least some of the radioactive isotopes, particularly with the trivalent radioactive ions. Semiresinous or non-resinous ion exchange substances which may be employed as the ionic substrata are, for example, sulphonated coal, polyphosphates, porphyrins, unmetallized phthalocyanines and other non-resinous chelating crystalloids.

There are also highly absorptive and adsorptive carrier materials that have little or no ion exchange qualities that can still be used to take up and hold radioactive substances in a solid condition. It is necessary to choose only those carrier materials to which radioactive elements or compounds can be affixed for a practical period of time without undue loss or dissolution of the radioactive substance from the carrier material. Thus some dehydrated gels, molecular sieves, molecular matrix silica gels and certain activated carbons are within the broad class of absorptive carrier materials which may be employed.

The suitability of a radioactive material for use in the powders of the present invention is determined primarily by its half-life. Radio-nuclides having half-lives of a million years or more are usually of little value because the specific activity or the number of millicuries per gram of the nuclide is too low. At the other extreme radionuclides with half-lives of less than one day are seldom of use unless they are the result of the continuous disintegration of a parent radionuclide of half-life longer than one day. In the latter case the parent radionuclide will for the purposes of this invention be considered to be the source of all of the radiations of the radionuclides resulting from it. With this relationship in mind all examples of radioactive materials for use in the paramagnetic radioactive powders of this invention have been drawn up in terms of the parent radionuclides only. Thus the mixture of the chlorides of cerium<sup>141</sup> and praseodymium<sup>141</sup> is considered to be simply cerium<sup>141</sup> chloride. Although most of the powerful radiations of the mixture come from the break up of praseodymium<sup>141</sup>, that isotope if separated from the cerium<sup>141</sup>, would be useful for only one hour or so because of its short half-life of 17.5 minutes.

The following list of radioisotopes which may be used is set down for the purposes of illustration only:

Isotope	Half-life	Isotope	Half-life
La <sup>140</sup>	40 hours	W <sup>185</sup>	73 days
Yt <sup>90</sup>	2.54 days	Ir <sup>192</sup>	74 days
Au <sup>198</sup>	2.7 days	Sc <sup>46</sup>	85 days
Sb <sup>122</sup>	2.8 days	S <sup>35</sup>	87 days
Au <sup>199</sup>	3.15 days	Ta <sup>182</sup>	115 days
Bi <sup>210</sup> (Ra E)	5.02 days	Ca <sup>45</sup>	163 days
Ba <sup>131</sup>	11.5 days	Ag <sup>110 m</sup>	270 days
Ba <sup>140</sup>	12.8 days	Ce <sup>144</sup>	282 days
Rb <sup>86</sup>	19.5 days	Cs <sup>134</sup>	2.3 years
Cr <sup>51</sup>	27.8 days	Pm <sup>147</sup>	2.6 years
Mixed fission products	—	Tl <sup>201</sup>	4 years
		Co <sup>60</sup>	5.3 years
Fe <sup>59</sup>	45 days	Eu <sup>152, 154</sup>	13 years
Hf <sup>181</sup>	45 days	Sr <sup>90</sup>	25 years
Sr <sup>89</sup>	53 days	Cs <sup>137</sup>	30 years
Sb <sup>124</sup>	60 days	C <sup>14</sup>	5568 years

Radionuclides of various half-lives may be divided arbitrarily into three groups according to half-life:

5 1. Long half-life isotopes (with half-lives of 2 years to 10,000 years)  
 2. Medium half-life isotopes (with half-lives of thirty days to two years)  
 10 3. Short half-life isotopes (with half-lives of a few hours to thirty days).

15 The long-lived isotopes of the first class are of great value in this invention because radioactive paramagnetic powders prepared from them lose radioactivity only at a very slow rate. The medium life of the second class may limit their usefulness. However, the class contains certain isotopes with desirable properties not found in the nuclides of the other two classes. For example, the cerium<sup>144</sup> 20 preparation as explained contains the extremely powerful praseodymium<sup>144</sup> and can be detected at long range. Also silver<sup>109m</sup> with a 270 day half-life may be converted easily from its ionic form to metallic silver 25 by reduction. The silver so fixed cannot be replaced by extraneous ions unless the silver is first oxidized back to the ionic state.

The short half-life isotopes have the disadvantage of deteriorating rapidly, but because they do lose their radioactivity in a matter of a few weeks or a few months they enable test bodies carrying paramagnetic radioactive powders containing these isotopes to revert back to an essentially nonradioactive condition by simple standing. For example paramagnetic radioactive powders containing gold<sup>198</sup> of half-life 2.7 days or yttrium<sup>90</sup> of half-life 2.54 days would retain only one-half of their original activity after 2.7 days, one sixteenth of their activity after 11 days and one 250th of their activity after 22 days. Retention of the test bodies carrying such particles for 44 days after testing would substantially eliminate the radioactivity by natural decay to sixteen millionths of the original activity.

30 A radioisotope is chosen not only according to whether or not it has a suitable half-life for the specific purpose, but also for the type of radioactive emission it has. For the most part the choice is between beta and gamma rays and between high energy and low energy emissions.

35 When gamma ray counting mechanisms are used, any isotope that gives off substantial

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quantities of gamma rays of energy above about 0.1 million electron volts (0.1 Mev), may be used if the other properties of valence, half-life and cost are suitable. The greater the number of nuclear changes per second which result in gamma rays the easier the detection of the treated magnetic particles will be, assuming gamma detection only. The chief advantage of using an isotope with energetic as opposed to weak gamma rays is that the counting instrument indicates more clearly the existence of the treated particles in the presence of the extraneous radiation making up the room background.

Particularly convenient counting instruments for gamma rays are scintillation counters. When these are used either singly or in pairs for coincidence counting, measurement of the gamma emitting isotopes is a simple matter.

When it is desired to reduce the amount of background radiation to extremely low levels, a well-type counter may be used which not only screens out most of the extraneous radiation from the room by the use of a lead shield, but registers a much higher percentage of the emitted rays from the test body. In a well counter the scintillation crystals can completely surround the body in a cylindrical manner while the photomultiplier tubes cemented directly to the crystal surfaces pick up a high percentage of the photons created in the crystals by the gamma rays from the test body.

Some radionuclides emit beta rays (electrons) only; some emit gamma rays only. However, most radionuclides together with their degradation products emit both beta and gamma rays. Therefore, either the beta rays or the gamma rays can be sensed or counted in these cases. It is somewhat less expensive to base a detection system on beta ray counting since the Geiger-Muller counter or a modification of it can be used rather than the more expensive scintillation counter. For example, caesium<sup>137</sup>, cobalt<sup>60</sup>, cerium<sup>144</sup> and silver<sup>110m</sup> which emit both types of rays are easily and conveniently detected and evaluated with a Geiger-Muller (GM) counter.

The same counter may be used, of course, for the isotopes which emit beta rays only. Thus thallium<sup>204</sup>, yttrium<sup>90</sup>, promethium<sup>147</sup>, strontium<sup>90</sup>, bismuth<sup>210</sup> and sulphur<sup>35</sup> are pure beta emitters. The use of a pure beta emitter has several advantages. First, the ratio of the isotope count to the room background can be made very high. Second, flaws on one side of a metal object can be detected separately from flaws on the other side of the object since the beta electrons generally do not pass through the object from the reverse side. Third, the detection operation can be observed by personnel from behind transparent plastic

shields without danger from the beta radiations of the test body.

As a further feature of the invention we provide a paramagnetic powder comprising particles composed of one or more paramagnetic particles and an ion-exchange resin or a chelating material adhesively bonded to said paramagnetic particle(s) by means of an adhesive, said ion-exchange resin or chelating material being adapted to chemically bond a radioactive material thereto by reaction therewith.

The following are examples, by way of illustration only, of specific paramagnetic powders and paramagnetic radioactive powders according to this invention and methods of using them:

#### EXAMPLE 1.

1362 Parts of ferromagnetic iron oxide (mainly  $Fe_2O_3$ ) powder are mixed thoroughly with 423 parts of an A stage epoxy resin made by reacting substantially one mol proportion of diphenylopropane with two mol proportions of epichlorohydrin. This and subsequent mixing operations are conveniently carried out in a water-jacketed horizontal kneading unit. 350 Parts of pulverized sulphonated styrene type cation exchange resin in its sodium form are added and the mixing continued until the viscosity drops markedly and mass is uniform. The material is removed from the mixer, cut into pieces about one cubic inch in size and allowed to stand at 25 to 35° C. for 16 to 18 hours. The lumps of aggregate are broken up finely enough to pass a 4 mesh screen (having an opening of 4760 microns). The resulting granular material is ground in a pebble mill with 1.05 times its weight of distilled water for 8 hours. The resulting slurry is allowed to settle and some particles of free ion exchange resin are levigated off. The solid is filtered off, dried at 105° C., then cured for one-half hour at 150° C. The cured ferromagnetic material is broken up again to form a loose powder.

One gram of the cured ferromagnetic material in 40 ml. of water is treated with one millicurie of the chloride of scandium<sup>46</sup> in 10 ml. of water, and the whole stirred for a few minutes. The aggregate is filtered off and washed with 40 ml. of water. The cake of aggregate contains almost 100% of the original scandium<sup>46</sup>. Additional washing does not remove any appreciable amount of scandium from the cake. The wet filter cake is dispersed in 750 ml. of water containing a nonfoaming wetting agent. The resulting slurry of radioactive ferromagnetic particles has a useful life of from three to six months depending upon the sensitivity of the detecting instruments used.

## EXAMPLE 2.

One gram of the cured ferromagnetic material of Example 1 in 20 ml. of water is treated with 0.8 millicurie of the chloride of promethium<sup>147</sup> in 10 ml. of water. The mixture is diluted with 470 ml. of water containing a wetting agent and a defoaming agent. As in Example 1 the radioactive ions are taken up virtually completely. The radioactive ferromagnetic particles of the suspension give off only beta rays having an average of 220,000 electron volts (0.22 m.e.v.) with no attendant gamma rays. Half of the original intensity is lost in 2.6 years.

## EXAMPLE 3.

One gram of the cured ferromagnetic material of Example 1 in 20 ml. of water is treated with 0.2 millicurie of a soluble salt of cerium<sup>144</sup> in 30 ml. of water. After stirring a few minutes, the radioactive magnetic particles may be freed of salt by filtering off and washing with distilled water or used directly by diluting with a suitable aqueous suspending medium. The particles give off extremely potent beta rays of 2.97 million electron volts. In addition the daughter isotope praseodymium<sup>141</sup> gives off 0.134 and 2.18 m.e.v. gamma rays. This composition is useful in those cases in which the detecting instrument must be located at a distance of a foot or more from the object to be examined.

## EXAMPLE 4.

40 Milligrams of metallic cobalt are subjected to a neutron stream from an atomic reactor until for example 0.001% to 0.005% of the cobalt is converted from cobalt<sup>59</sup> to cobalt<sup>60</sup>. The cobalt metal so treated is dissolved in a suitable acid such as nitric acid. The resulting solution is diluted to 50 ml. and mixed with a suspension of 5 grams of cured ferromagnetic material of Example 1 in 50 ml. of water. After a few minutes the aggregate particles are filtered off, washed with water and dried. With due precautions to avoid loss, 5 grams of the dry material so formed is dispersed in twice its weight of mineral oil to form a thin paste. The paste is further dispersed in 2500 ml. of kerosene. The result is a suspension in kerosene of radioactive ferromagnetic particles suitable

for use with test pieces which might rust if tested with aqueous test suspensions.

## EXAMPLE 5.

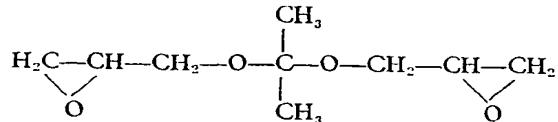
125 Grams of the cured ferromagnetic material of Example 1 are mixed with 50 grams of a siliceous filter aid and wet down with enough distilled water to form a free-flowing slurry. A conventional vertical ion exchange column having a filter disk at the bottom is filled three-quarters full with the slurry. The acid solution of cobalt nitrate similar to that used in Example 4 is then introduced into the top of the column at the rate of 10 ml. per minute. The flow of liquid from the bottom of the column is adjusted to the same rate. After the solution has all been added, distilled water is run down through the column until the effluent is substantially free from radioactivity. The contents of the column are removed in the wet state and the radioactive ferromagnetic particles separated from the filter aid by any suitable means such as levigation or selective wetting of the radioactive particles by a water-insoluble organic liquid of relatively low viscosity such as Stoddard Solvent.

## EXAMPLE 6.

1 Millicurie of caesium<sup>137</sup> chloride with a half-life of approximately 30 years containing small amounts of barium<sup>137</sup> chloride, the daughter radionuclide, with a half-life of 2.6 minutes, is treated with 10 ml. of water until complete solution results. One-half gram of the cured ferromagnetic material of Example 1 in 20 ml. of water is mixed with the caesium chloride solution, the whole stirred for several hours and the solid particles filtered off. After washing the cake with water, the cake and the filtrate are examined in similar volumes of water to determine the relative activity. 40% of the activity is found to be retained by the solid material so that the latter has a specific activity of 0.8 milli-curries per gram. A suitable dispersing agent may be used if desired in the suspending liquid.

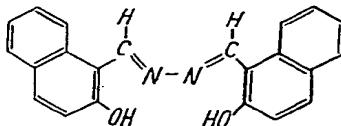
## EXAMPLE 7.

1362 Parts of ferromagnetic iron oxide powder ( $Fe_3O_4$ ) are mixed thoroughly with 423 parts of a substantially monomeric epoxy resin intermediate having the formula:



175 parts of pulverized sulphonated styrene type cation exchange resin are added. 175

parts of the fluorescent azine of 2-hydroxy-1-naphthaldehyde of the formula:



are also added. The above ingredients are kneaded until thoroughly mixed. 63.4 parts of melted *m*-phenylenediamine are added and 5 the mixing continued. The mass is allowed to stand overnight at 25 to 40° C. The material is then ground in a pebble mill with water for 8 to 10 hours. The slurry is filtered and the cake is mixed with water to produce a 10 paste or slurry of 50% solids content. The ferromagnetic aggregate particles of the slurry are highly fluorescent. They glow with a distinctive greenish yellow colour under ultra-violet light. Instead of the above specific 15 fluorescent azine, numerous other satisfactory fluorescent materials may be employed, such as, for example, the azine of 2-aceto-1-naphthol, fluorescent zinc oxide, fluorescent or phosphorescent zinc sulphides, Lumogen 20 L Red Orange and various resinous type fluorescent pigments such as those described in United States Patent No. 2,498,592. Further, it is not necessary that the fluorescent agent, if a dye rather than a pigment, be 25 bonded to the ferromagnetic element by the resin binding the carrier component thereto. Instead, as one energy-releasing indicating medium which may be used together with radioactive material as another energy-releasing indicating medium, a fluorescent dye 30 ionized in a solution may be bonded to the testing particles by immersion of the powders therein. Such solution may be the same solution in which the radioactive material is dissolved so that both are bonded to the testing 35 particles simultaneously or the radioactive material and the fluorescent dye may be attached by successive immersion in separate solutions.

40 One gram of the above 50% paste is added to 15 ml. of water. 0.4 millicurie of thallium<sup>204</sup> nitrate in 15 ml. of water is mixed in. After one hour the solid is filtered off and dried if desired. The solid retains approximately 45% of the radioactive ions. The 45 solid is suspended in any suitable liquid such as water, organic solvents or petroleum derivatives stable to radiation. The particles containing thallium<sup>204</sup> give off powerful beta radiation of 0.765 m.e.v. No gamma rays are emitted. This radioactive product may be used to advantage in large amounts in those cases in which it is desired that personnel remain 50 in close proximity to a flaw detection system in which large test pieces are dipped or sprayed with the slurry provided only that plastic or glass shields, for example, are interposed between the system and the personnel,

60 since the beta rays cannot pass through such shields. The half-life of the thallium product is 4 years. After automatic sorting of the parts by radiation detecting counters, parts rejected for high activity may be inspected further by personnel using ultra-violet light since any indications on the test pieces are 65 fluorescent as well as radioactive.

#### EXAMPLE 8.

An adsorptive aggregate is made up as in Example 7 except that 350 parts of pulverized activated bauxite are used in place of the 70 pulverized cation exchange resin.

0.5 Millicurie of sulphur<sup>35</sup> in the form of sulphuric acid is taken up in 10 ml. of water and mixed with one gram of this adsorptive aggregate. The resulting radioactive ferromagnetic particles are filtered off, washed with water and dried. They are suspended in a gas oil having a boiling range just above that of kerosene. The radioactive component has a half-life of 87 days. The beta rays given off have an average energy of 0.167 m.e.v.

#### EXAMPLE 9.

An adsorptive aggregate is made up as in Example 1 except that 1362 parts of bright iron filings passing a 150 mesh screen (having openings of 104 microns) are used in place of the iron oxide powder.

This product is treated with a solution of caesium<sup>137</sup> chloride to produce a ferromagnetic radioactive aggregate.

#### EXAMPLE 10.

1300 Parts of ferromagnetic iron oxide (mainly Fe<sub>2</sub>O<sub>3</sub>) powder are mixed with 400 parts of a substantially monomeric epoxy resin intermediate containing two epoxide rings per molecule. 350 parts of air-dried anion exchange resin of the quaternary ammonium-styrene type in its chloride form are added. The ingredients are kneaded together at room temperature. 60 Parts of melted *m*-tolylenediamine are added and the mixing continued until the mass is uniform. The mass is allowed to stand at 25 to 35° C. overnight, then broken up into particles of a size suitable for pebble milling. The material is ground in a pebble mill with an equal weight of deionized water for 6 hours. The resulting slurry is filtered off and air dried at room temperature. The dry filter cake is age-cured by holding it at 40-45° C. for three weeks. The cake is then broken up to form a loose powder; for example, by grinding for 20 minutes in a pebble mill with an equal weight of water. This material is manufactured at temperatures near room temperature because the quaternary ion exchange resin breaks down if subjected to temperatures above about 45° C. The resulting material may be stored in the form of the 50 percent slurry produced by milling.

0.5 Millicurie of a solution of sulphuric acid derived from radioactive sulphur<sup>35</sup> is brought to a volume of 20 ml. and mixed with two grams of this final 50 percent slurry. 5 The radioactive sulphate radicals are taken up to the extent of practically 100 percent by the quaternary ion exchange centres of the aggregate particles. The hydrochloric acid formed and any sulphuric acid not taken up 10 are removed by filtering off the solid and washing with water. The resulting product is dispersed in a suitable suspending medium.

EXAMPLE 11.

15 1 Millicurie of tantalum<sup>182</sup> in the form of potassium tantalate in 20 ml. of water was treated with one gram of the 50 percent slurry of Example 10. The radioactive ion is taken up by the particles to the extent of more than 80 percent. The radioactive half-life of 20 the product is 115 days.

EXAMPLE 12.

One gram of the ferromagnetic material of Example 7 is dispersed in 40 ml. of water. 25 0.5 Millicurie of freshly prepared gold<sup>193</sup> chloride,  $\text{AuCl}_3$ , in acid solution is diluted up to 20 ml. and mixed in. The radioactive ions are completely absorbed by the particles of fluorescent aggregate. The slurry may be diluted directly with water containing a 30 wetting agent and a defoaming agent. The half-life of the gold<sup>193</sup> is 2.7 days.

EXAMPLE 13.

One gram of the ferromagnetic material of Example 7 is dispersed in 30 ml. of water. 35 0.5 Millicurie of radium chloride in 15 ml. of water is mixed in. The solid is filtered off, washed with distilled water and made up to form a working suspension with additional water and a wetting agent. Alpha, beta, and 40 gamma rays are emitted by the radium and its daughter radionuclides.

EXAMPLE 14.

An adsorptive ion exchange aggregate is made up as in Example 7 except that in place 45 of the sulphonated styrene type resin an equal weight of a phenol-sulphonic acid-formaldehyde type ion exchange resin is employed.

One gram of this ion exchange aggregate in 30 ml. of water is treated with one milli-curye of caesium<sup>137</sup> chloride in 10 ml. of water. The solid is filtered off and washed with water. The cake is dispersed in a liquid capable of wetting both the particles and metal test objects.

EXAMPLE 15.

1360 Parts of ferromagnetic iron oxide powder ( $\text{Fe}_2\text{O}_3$ ) are mixed thoroughly with 455 parts of 3,4-epoxy-6-methylcyclohexylmethyl - 3,4 - epoxy - 6 - methylcyclohexane carboxylate. 350 Parts of a pulverized carboxylic type ion exchange resin made by copolymerizing acrylic acid and divinyl benzene are mixed in. 108 Parts of diglycolic acid are added and the whole mixed in a 60 kneading unit for 1 hour until the mass starts

to harden. The mass is broken up, spread on a tray, and allowed to stand overnight. The product is cut or ground into small pieces, then ground with 1.1 times its weight of de-ionized water for 20 hours. The solid material is filtered off and dried at 80° C. The powder is cured for one-half hour at 145° C. The cured material is ground for a short time with 1.2 times its weight of water and filtered off on a large Buchner funnel. The cake is redispersed in 7 litres of water and converted from the carboxy form to the sodium carboxylate form by stirring the suspension and adding very slowly 1.4 litres of 2.5 normal sodium hydroxide solution. The whole is allowed to stand 24 hours, then filtered and washed with 14 litres of de-ionized water. The resulting cake is made up to a solids content of 50% by adding water.

2 Grams of this 50% cake are mixed with 400 ml. of water and treated with 0.4 milli-curye of an aqueous solution of a salt of cobalt<sup>60</sup>. The ion exchange reaction causes substantially 100% of the cobalt<sup>60</sup> ions to replace sodium attached to the carboxylate groups of the ion exchange resin. Although the carboxylate groups hold the cobalt ions very strongly the particles may be freed of radioactive cobalt by washing with dilute mineral acids. This capability of ion exchange components to be stripped of the attached metal ions by acid or in many cases by salts such as sodium chloride can be utilized in various decontamination procedures.

EXAMPLE 16.

The 50% cake of Example 15 is treated with a mixture of the chlorides of europium<sup>152</sup> and europium<sup>154</sup>, the first of which has a half-life of 13 years, the second of 16 years, to give suspension of ferromagnetic radioactive particles having a specific activity in milli-curies per gram of aggregate between 0.1 and 2.0.

EXAMPLE 17.

Resinous material of the styrene-divinyl benzene type carrying substituents having the structure  $-\text{CH}_2-\text{N}(\text{CH}_2-\text{COOH})_2$  is ground to a powder. 350 Parts of this powder are kneaded with 1362 parts of ferromagnetic iron oxide ( $\text{Fe}_2\text{O}_3$ ) and 423 parts of a glycidyl type epoxy resin intermediate containing two epoxide rings per molecule. 63.4 Parts of melted *m*-phenylenediamine are added and the mixing continued until the mass is uniform. The mass is allowed to convert to the B stage at room temperature. It is broken up and pebble milled with 1.05 times its weight of distilled water for 8 hours. The resulting material is allowed to settle and the ferromagnetic particles separated and dried. The dry material is kept at 40° C. for three weeks. The cured material is ground for a short time with 1.1 times its weight of water and filtered. The cake is redispersed in water and treated with a quantity of sodium

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hydroxide corresponding to one molecule of NaOH for each carboxyl group present in the crude material. The product is thus partially converted to its sodium salt form. The solid is filtered off and washed with distilled water. The cake is made up to a solids content of 50%.

2 Grams of this 50% slurry are stirred into 40 ml. of water. 0.5 Millicurie of cobalt<sup>60</sup> nitrate exhibiting a specific activity of 10,000 millicuries per gram of cobalt is diluted with a few ml. of water and added to the dilute slurry. The cobalt ions are very strongly sequestered and held by the ionized di-(carboxymethyl)amino groups of the aggregate.

EXAMPLE 18.

2 Grams of the 50% slurry of Example 17 are mixed with 40 ml. of water. 0.5 Millicurie of cerium<sup>144</sup> chloride in 20 ml. of water is added with stirring. The solid is filtered off, dried, milled into twice its weight of petrolatum and diluted with a relatively non-volatile liquid hydrocarbon or halogenated hydrocarbon.

EXAMPLE 19.

Two clean three inch steel turbine blades, one known to have almost invisible cracks in its surface, the other free of cracks, are magnetized between the poles of a powerful magnet. Both blades are dipped in the radioactive slurry of Example 1 then examined with a Geiger Muller counter. With a room background radiation count of 120 counts per minute, the uncracked three inch blade shows a total count of 275 counts per minute three inches from the counter while the cracked blade shows a total count of 425 counts per minute at the same distance. Accordingly, the counts adjusted for background count to 155 and 305, respectively, show an increase of some 100% attributable to radioactive particles deposited on the flaws in the cracked blade. The blades are cleansed of all radioactive particles, preferably by vibration in contact with a suitable liquid in an ultrasonic cleaning unit.

EXAMPLE 20.

A 0.4% carbon steel bar is heated to red heat, quenched in a spray or bath of water, then reheated to a dull red heat and re-quenched so that quenching cracks form in the surface of the bar. The surface is cleaned to remove scale. The bar is clamped between two electrodes of a magnetizing unit and a current of 1000 amperes pass through the bar for one-third of a second. The bar is removed and the slurry of Example 6 in a uniform condition of agitation is flowed over the bar. The radioactive ferromagnetic particles adhere to the bar predominantly in those areas which have quenching cracks. An unquenched bar is similarly magnetized and treated with the slurry. Both pieces are examined with a Geiger Muller counter for radioactivity. It is

found that the unquenched bar shows a relative count of 800, not including the room background of 700 counts per minute, while the quenched bar shows a count depending on the number and size of the cracks of from 1000 to 1150 counts per minute, not including the room background. The relatively high radioactivity of the unquenched bar may be reduced very largely by a water dip as in Example 21.

Similar readily evident differences in the radioactivity of flawed and unflawed test bodies will be observed by repeating the tests of Examples 19 and 20 with the remaining radioactive magnetic powders of Examples 1 to 18. For production testing of numerous duplicate parts using known flawed and unflawed parts as controls and taking into account possible deterioration of radioactive materials of short half-life, background radiation, et cetera, count ranges for acceptable, questionable, and rejectable parts may be established.

EXAMPLE 21.

A plain unquenched magnetized steel bar and a strongly quenched and magnetized steel bar, similar to those used in Example 20, are dipped in the stirred suspension of Example 2. Each piece is then rinsed to remove loose adhering particles by dipping in water with mild agitation. With a room background of 300 counts per minute the plain piece shows a total count of 400 counts per minute, the quenched piece with cracks shows a total count of 650 counts per minute, an increase of 250% if the background is subtracted.

EXAMPLE 22.

A plain unquenched magnetized steel bar and a strongly quenched and magnetized steel bar which has large cracks on a side designated as the front and which has a few small cracks on the other side designated as the back are dipped in the stirred suspension of Example 3. Each bar is then rinsed to remove loosely adhering particles. The plain piece shows a count of 1300 including a room background of 950 counts per minute. The back of the quenched piece shows a count of 1450 or 150 more than the plain piece. The front of the quenched piece shows a count of 1800 or 500 more than the plain piece under the same conditions. Thus with the suspension of Example 3, it is possible not only to detect the presence of flaws in the steel bar, but also to distinguish between the side of the bar that has large cracks from the side of the bar which has small cracks.

EXAMPLE 23.

A 0.4% carbon steel bar 12 inches long is heated to red heat for a distance of three inches at one end and quenched with water. The bar is reheated over the same three inch length to red heat and quenched a second time so that cracks develop at the quenched end. The bar is cleaned to remove loose scale.

5 The bar is dried, magnetized by the use of a surrounding coil and flooded with the kerosene suspension of cobalt<sup>60</sup> impregnated particles of Example 4. The bar is rinsed with plain kerosene. The bar is scanned with a Geiger Muller counter from one end to the other holding the counter at a distance of one inch from the bar. The cracked end of the bar shows a disintegration count significantly higher than the uncracked end. Instead of scanning the bar by relative movement of the sensing instrument and the bar, the bar or other object may be brought into position with respect to a number of sensing elements which are stationary with respect to each other, and the number of counts per minute produced by each sensing element compared in order to locate the position of flaws on the test object.

20 EXAMPLE 24.

25 A 0.4% carbon steel bar is heated and quenched so as to produce definite cracks in the surface. The bar is magnetized and dipped in a suspension containing the product of Example 6 then rinsed with water. The bar shows a Geiger Muller count of 900 counts per minute not including the room background. The bar is then dipped in 10% solution of sodium chloride for one minute and mildly agitated. The sodium ions of the solution replace the cesium ions attached to the sulphonate groups of the ion exchange resin. The bar removed from the salt solution shows a count of 200 counts per minute not including room background. A second one minute dip in fresh 10% salt solution reduces the radioactivity of the bar to 50 counts per minute not including background. Thus, the two dips brought about a reduction of the radioactivity of the bar of 93%.

30 EXAMPLE 25.

35 Employing radioactive fluorescent magnetic particles made according to Example 7, large production quantities of repetitive parts are magnetized, subjected to a flush with a dispersion of fluorescent, radioactive ferromagnetic powders, suitably drained and rinsed, and then conveyed by automatic conveyor past suitable counters. Unflawed parts 40 pass the counters, without activating any signal or sorting device, to a suitable receiving bin for such subsequent decontaminating as may be desired. Should the count of any additional piece exceed the established "safe" limit, a signal actuated by the counter may 45 stop the conveyor or actuate a sorting device. Either by examination under filtered ultraviolet light at that moment or later, the operator, shielded to whatever degree is required, may ascertain from the visible indication of the fluorescent powdered material the extent and location of the flaws in the piece 50 being tested. If the article is to be removed from the line or directed to a separate line for closer inspection the particles may be fixed

55 by means of a clear lacquer spray to avoid dusting off. Visual inspection can minimize "over-inspection" of acceptable parts due to false or irrelevant flaw indication. Such false or irrelevant indications can be caused in a 60 number of ways. For example, often the contour and intended use of the part is such that, a flaw may be in a sufficiently uncritical or relatively unstressed portion of the part to 65 permit it to be accepted, whereas a flaw of the same extent in a critical area would be a cause for rejection. Also, individual pieces 70 may give false indications by accumulating and holding excess quantities of the radioactive powder for reasons other than the presence of flaws, i.e., because of grease or cutting oils not properly cleaned, or unremoved flash (in the case of forgings), excessive surface roughness (in the case of castings) etc. The visual supplemental inspection 75 afforded by fluorescent radioactive test powders can usually enable the operator to distinguish readily between true and false 80 flaw indications. In any event, due to automatic pre-selection before visual inspection, the operator is relieved of the chore of 85 visually inspecting numerous acceptable pieces, the number of operators required is greatly reduced, and the chance for human 90 error is greatly minimized by eliminating the boring task of careful visual inspection of 95 clearly acceptable items in addition to the rejectable and questionable ones in a given production lot.

95 WHAT I CLAIM IS:—

100 1. A paramagnetic radioactive powder comprising particles composed of one or more paramagnetic (as herein defined) particles, a carrier material for a radioactive material adhesively bonded to said paramagnetic particle(s) by means of an adhesive and a radioactive material bonded to an exposed surface of said carrier material by physical or chemical bonds.

105 2. A powder as claimed in claim 1 in which the paramagnetic particles are ferromagnetic.

110 3. A powder as claimed in claim 1 or claim 2, in which the carrier material is bonded to the paramagnetic particles by means of a synthetic resin.

115 4. A powder as claimed in claim 1 or claim 3, in which the paramagnetic particles are particles of carbonyl iron, aluminium, or red or black iron oxide.

120 5. A powder as claimed in any of the preceding claims in which the carrier material is a chelating or ion-exchanging material.

125 6. A powder as claimed in claim 5, in which the carrier material is an ion-exchange resin.

130 7. A powder as claimed in claim 6, in which the carrier material is a cation-exchange resin, and the radioactive material is a radioactive position metal ion the emission energy

of which is in excess of 50,000 electron volts.

8. A powder as claimed in claim 7, in which the cation-exchange resin is a sulphonated cross-linked polystyrene resin, a formaldehyde-phenol carboxylic acid resin, a formaldehyde-phenol sulphonic acid resin, a cross-linked polyacrylic acid resin, or a substituted cross-linked polystyrene resin in which the substituents on the benzene rings are di-(carboxymethyl)-aminomethyl groups.

9. A powder as claimed in claim 7 or claim 8, in which the radioactive positive metal ion is derived from  $Cs^{137}$ ,  $Co^{60}$ ,  $Tl^{204}$ ,  $Pm^{147}$ ,  $Ce^{144}$ ,  $Eu^{152}$ ,  $Eu^{154}$ ,  $Sc^{46}$ , or  $Au^{198}$ .

10. A powder as claimed in claim 6, in which the carrier material is an anion-exchange resin, and the radioactive material is a radioactive negative ion the emission energy of which is in excess of 50,000 electron volts.

11. A powder as claimed in claim 10, in which the anion-exchange resin is a quaternary ammonium derivative of a cross-linked polystyrene resin.

12. A powder as claimed in claim 10 or claim 11, in which the radioactive negative ion is one derived from sulphur<sup>35</sup> or tantalum<sup>182</sup>.

13. A powder as claimed in any of claims 3 to 12, in which the synthetic resin bonding the paramagnetic particles to the radioactive material is an epoxy resin.

14. A powder as claimed in any of the preceding claims in which at least a substantial proportion of the particles of the powder also contains a visible light-emitting fluorescent material and/or a colouring material.

15. A powder as claimed in claim 14, in which the particles of the powder include a fluorescent dye or pigment adhesively bonded to the paramagnetic particles.

16. A powder substantially as herein described with reference to any of Examples 1 to 18.

17. A process for the production of a paramagnetic radioactive powder as claimed in claim 1, in which pulverulent paramagnetic particles and a carrier material for a radioactive material are dispersed in a liquid synthetic resin, the liquid synthetic resin is solidified to bond paramagnetic particles and carrier material, the solidified product is ground to yield a fine paramagnetic powder composed of paramagnetic particles and carrier material bonded together, and the fine paramagnetic powder is disposed in a fluid dispersion or solution of radioactive material so that a proportion of the radioactive material becomes attached by physical or chemical bonds to the carrier material.

18. A process as claimed in claim 17, in which the paramagnetic radioactive powder produced is separated from the fluid contain-

ing radioactive material and dispersed in a liquid inert to the paramagnetic radioactive powder and to any article to be tested.

19. A process as claimed in claim 17 or claim 18, in which a fluorescent dye or pigment is dispersed with the paramagnetic particles and carrier material in the liquid synthetic resin.

20. A process as claimed in claim 17 or claim 18, in which the paramagnetic radioactive powder is dispersed in a solution containing a dissolved ionized fluorescent dye.

21. A method of testing magnetizable test bodies for flaws comprising the steps of creating a magnetic flux with a first control test body known to contain no magnetically revealed flaws, flushing the first control test body with a dispersion of paramagnetic radioactive powder as claimed in claim 1, and measuring the radioactivity of the first control test body after the powder has been substantially removed from the unflawed surfaces of the first control test body to establish a range of permissible radiation from the first control, repeating these steps with respect to a second substantially duplicate control test body known to be sufficiently flawed to render it unsuitable for service in order to establish a level of radiation which, if exceeded, would indicate that the test body is unsuitable for service, and repeating the steps with respect to substantially duplicate test bodies of unknown flawed condition to ascertain whether sufficient radioactive magnetic powder has been retained on the surface of the test bodies in the area of flaws to indicate the probable unserviceability of said test bodies.

22. A method as claimed in claim 21 including the further step of deactivating any radioactive powder which may remain on the test bodies by removal of the radioactive component.

23. A method as claimed in claim 21 or claim 22, in which the paramagnetic radioactive powder contains a fluorescent dye or pigment, including the further step of then inspecting test bodies of questionable serviceability under fluorescogenous radiation to ascertain visually the extent and location of flaws which may exist, the existence of such flaws being indicated by the emission of visible light by the fluorescent dye or pigment in the powder.

24. A method of testing magnetizable test bodies for flaws substantially as herein described with reference to any of Examples 19 to 25.

25. A paramagnetic powder comprising particles composed of one or more paramagnetic particles and an ion-exchange resin or a chelating material adhesively bonded to said paramagnetic particle(s) by means of an adhesive, said ion-exchange resin or chelating

material being adapted to chemically bond a radioactive material thereto by reaction therewith.

5 26. A powder as claimed in claim 25, in which the paramagnetic particles are particles of carbonyl iron, aluminium, or red or black iron oxide.

27. A powder as claimed in claim 25 or

claim 26, in which the powder particles include a fluorescent dye or pigment bonded to the paramagnetic particles. 10

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Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press (Leamington) Ltd.—1963. Published by The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.